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FAILURE ANALYSIS OF EPOXY-LINED ASBESTOS-CEMENT PIPE.(U)

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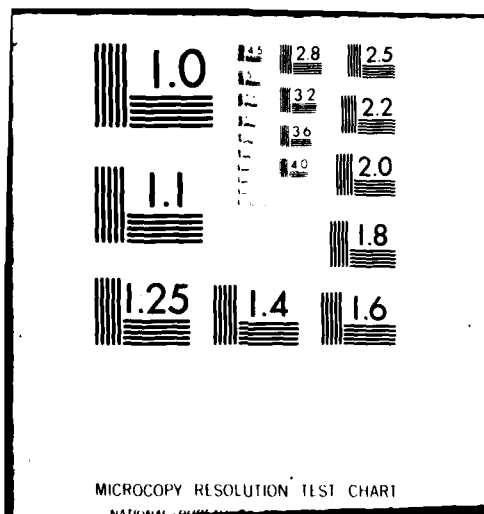
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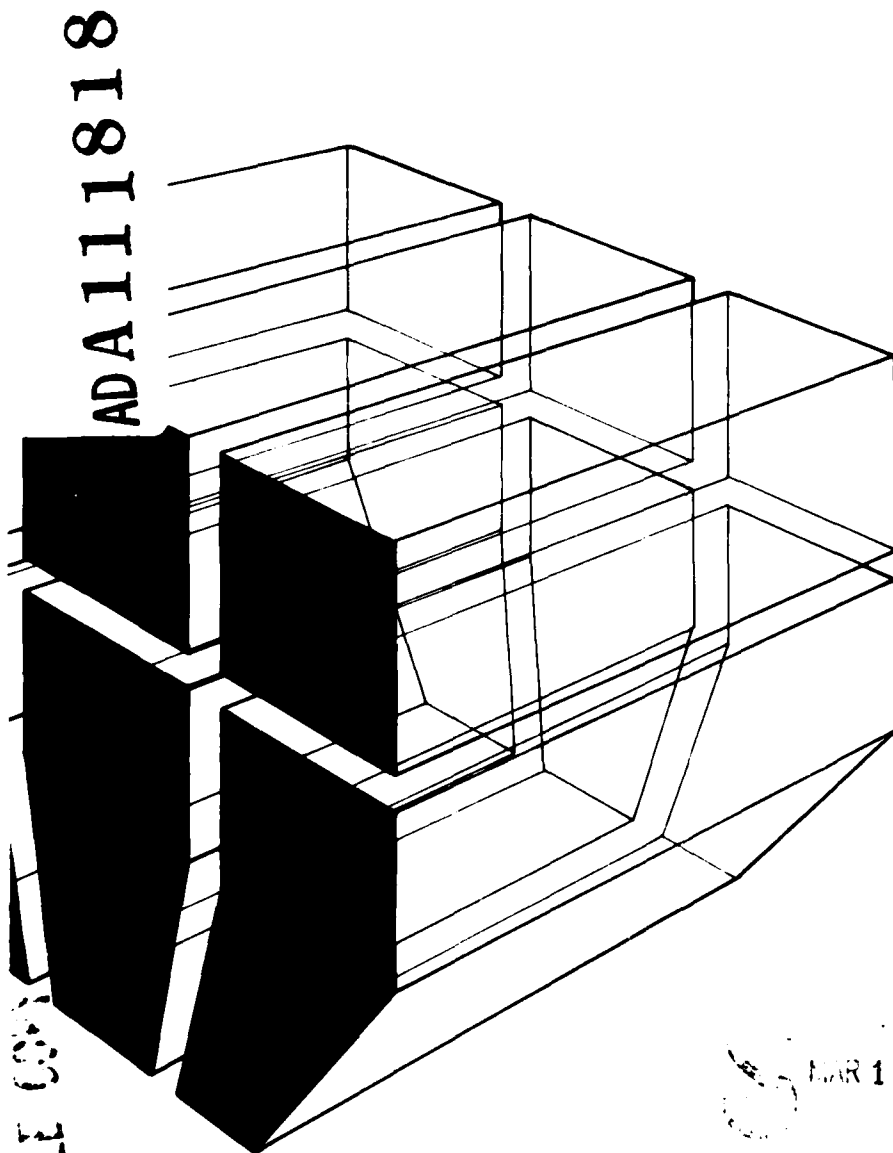


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Technical Report M-304  
December 1981

FAILURE ANALYSIS OF EPOXY-LINED  
ASBESTOS-CEMENT PIPE

by  
Alvin Smith



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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br><br>This report presents the findings of an investigation into the causes of failure or epoxy lining in asbestos-cement sewer pipe used in certain Middle East construction projects.<br><br>The asbestos-cement pipe passed all tests performed on it to see whether it met the original specifications. The epoxy lining did not meet the requirements in some samples. |                                      |   |

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The report concludes that the lining failures occurred because of the following factors:

1. Poor proportioning of curing agent to epoxy resin, mixing, and curing, which led to residual stresses, large amounts of swelling, softening, and strength loss both internally (cohesively) and at the adhesive bond (adhesively).
2. Environmental effects (high soluble salt content of the soil), and possible aggressiveness of the materials carried in the pipe.

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## FOREWORD

This investigation was conducted by the Engineering and Materials Division (EM), U.S. Army Construction Engineering Research Laboratory (CERL), for the Middle-East Division (MED) (Rear), U.S. Army Corps of Engineers. Funds were provided by Military Interdepartmental Purchase Request Number W26WKS10001. The MED-Rear coordinator was Mr. Don Slater, MEDED-F.

Some highly specialized evaluations were contracted to and performed by Dr. Richard Wool, Polymer Group, University of Illinois, and his assistance is acknowledged. Appreciation is expressed to LT O. S. Marshall, Jr. of the Polymer Applications Team, EM Division for the many tedious tests he performed, interpretations of results, and the suggestions he offered.

Dr. R. Quattrone is Chief of EM, COL Louis J. Circeo is Commander and Director of CERL, and Dr. L. R. Shaffer is Technical Director.



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# FAILURE ANALYSIS OF EPOXY-LINED ASBESTOS-CEMENT PIPE

## 1 INTRODUCTION

### Background

Sewer force mains, gravity sewer lines, and insulated chilled-water lines of epoxy-lined asbestos-cement pipe were installed in some coastal areas in the Middle East in 1978 and 1979. This type of pipe was apparently selected based on potential corrosion problems of buried metal pipes due to the composition of the soil and soil water in the area.<sup>1</sup>

Failures of the epoxy lining were first noted in June 1980; fragments of the lining material were found in the filter of a sewer line. An investigation of all three kinds of service pipe showed varying degrees of lining blistering and sloughing off. No specific pattern relating failure either to kind of service or length of service could be found. In fact, some of the failures had occurred in installed pipe that had been used very little or not at all. Unused, uninstalled pipe in pre-construction stockpiles in the area showed no lining failure.

### Objectives

The objectives of this study were: (1) to determine whether the epoxy-lined asbestos-cement pipe met the specifications under which it was procured, (2) to analyze the causes of failure of the epoxy lining in the installed piping systems, and (3) to recommend solutions to the problem.

### Approach

Standard American Society for Testing and Materials (ASTM) tests were used to evaluate how well the asbestos-cement pipe and the epoxy-lining materials conformed to specifications. In addition, special tests and techniques were used to determine the causes of adhesion failure, blistering, and fracture of pieces of lining from the asbestos-cement substrate.

<sup>1</sup> Study of Corrosive Soil Conditions and Need for Cathodic Protection, Various [Middle East] Locations, Delivery Order 0005, Contract DACA 75-76-D-0040 (Dames and Moore, August 1976).

### Scope

The study was limited to checking the compliance of the asbestos-cement pipe with original specification requirements and determining the cause of failure of the epoxy lining, taking into account both internal and external environmental factors as well as material properties and the manufacturers' procedures.

## 2 DESCRIPTION OF MATERIALS AND SPECIFICATIONS

### Asbestos-Cement Pipe

Asbestos cement pipe is composed of asbestos fibers (15 to 20 percent), silica (32 to 34 percent), and Portland cement (48 to 51 percent). The asbestos fibers are milled, cleaned, and mixed with the silica and cement. Following a thorough blending procedure, water is mixed with the blend and a felt is produced on the surface of fine mesh screen drums. The felt is wound off the drums and onto a pipe mandrel where it is built up in layers, under pressure, until the required thickness is reached. Excess water is removed during the felt transfer, and hydraulic pressure is used to densify the pipe material. The "green" pipe is then partially cured; the mandrel is removed and a final curing process carried out in a steam autoclave. After curing, the pipe is trimmed and any subsequent secondary operations are performed. The entire pipe-making process has been described by Cohn.<sup>2</sup>

The quality of asbestos-cement pipe is usually verified by testing, as prescribed in various ASTM Standard Specifications.<sup>3</sup> Included in quality assurance testing are composition, hydrostatic strength, flexural strength, crushing strength, chemical requirements, sizes and dimensions, and workmanship and finish. Test procedures are detailed in ASTM C500.<sup>4</sup>

<sup>2</sup> Morris M. Cohn, *Sewers for Growing America* (Certainteed Productions Corporation, Ambler, PA, 1966) p 138 ff.

<sup>3</sup> ASTM C296-78, "Asbestos-Cement Pressure Pipe," Part 16 (American Society for Testing and Materials [ASTM], 1978); ASTM C428-78, "Asbestos-Cement Nonpressure Sewer Pipe," Part 16 (ASTM, 1978); ASTM C644-78, "Asbestos-Cement Nonpressure Small-Diameter Sewer Pipe," Part 16 (ASTM, 1978).

<sup>4</sup> ASTM C500, "Testing of Asbestos-Cement Pipe," Part 16 (ASTM, 1978).

Two types of asbestos-cement pipe are available; they differ in uncombined calcium hydroxide content. Type I may have more than 1 percent uncombined calcium hydroxide; Type II must contain less than 1 percent uncombined calcium hydroxide. The amount of uncombined calcium largely determines resistance to attack by soluble sulfates in soil water.

#### Epoxy Lining

In some cases, asbestos-cement pipe is lined with an epoxy material—especially when the substance to be carried is abrasive or highly corrosive. This protects the pipe from the effects of its contents.

The epoxy used for lining the pipe must be 100 percent solids (no solvent), applied and cured with a minimum thickness of 12 mils. ASTM C541 gives test requirements.<sup>5</sup>

The method of applying the liquid epoxy lining is proprietary. The pipe is rotated about its long axis during and after application of the lining material until the epoxy gels and cures. Exposure of the system to heat speeds up curing, thus the pipe must be rotated during the entire process. In essence, the lining is centrifugally cast in place within the pipe. Full cure of the epoxy system used for asbestos-cement pipe lining is usually judged by hardness. A Barcol hardness of 40 or higher signifies adequate cure.

#### Specimens Provided

A total of seven specimen sets of epoxy-lined asbestos-cement pipe were provided for testing. Each set consisted of two pieces of pipe, each 12-in. (304-mm) long, one piece of pipe 6-in. (152-mm) long, and three broken pipe fragments about 2 in. by 6 in. (50 mm by 152 mm). Table 1 describes the pipe specimens.

Each pipe section was recut so that the length could be measured accurately and the ends could be made smooth and perpendicular to the pipe length.

Two soil specimens were provided. Both contained silt, sand, and gravel. Both soils were wet when received.

CERL was provided one water sample from the chilled water system. The Corps of Engineers Middle East Division (Rear) (MED[Rear]) sent a second water sample (from the sewer system) elsewhere for tests. (Test results of the second water sample have not been received and will not be included in this report.)

<sup>5</sup>ASTM C541, "Linings of Asbestos-Cement Pipe," Part 16 (ASTM, 1978).

**Table 1**  
**Pipe Specimens**

| Specimen Set Number | Diameter, In. (mm) | Intended Use  | History    |
|---------------------|--------------------|---------------|------------|
| 1                   | 10 (254)           | chilled water | in-service |
| 2                   | 10 (254)           | chilled water | original*  |
| 3                   | 6 (152)            | force main    | in-service |
| 4                   | 8 (203)            | force main    | original*  |
| 5                   | 8 (203)            | gravity       | in-service |
| 6                   | 8 (203)            | gravity       | original*  |
| 7                   | 6 (152)            | sewer**       | original*  |

\*Unused, from on-site stockpile

\*\*From a different manufacturer

### 3 TEST AND EVALUATION

#### Pipe Tests

##### Crushing Strength

Tests were performed on one 12-in. (305-mm) length of each pipe specimen set in accordance with Section 11.1.1 of ASTM C500. Table 2 describes the requirements and results of the tests. Figure 1 shows the test setup.

Summary of Results: all in-service and original pipe specimens tested exceeded the crushing strength minimum requirements.

##### Sizes and Dimensions

The inside diameters of each of the three pipe sections in each specimen set were measured with a vernier caliper accurate to 0.001 in. (0.025 mm). The largest and smallest diameter at one end of each pipe was recorded, and the difference between the nominal pipe diameter and the measured diameter was noted. Table 3 shows the size and dimension results. The standard length could not be determined since only short pieces of pipe were provided for the tests.

Summary of Results: all specimens were within the diametral requirements.

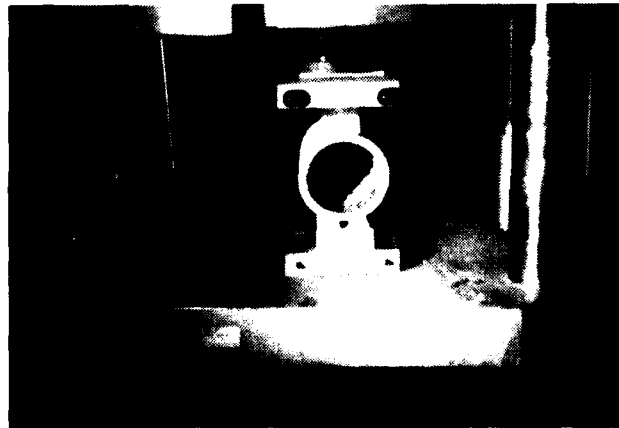
##### Uncombined Calcium Hydroxide

Uncombined calcium hydroxide was determined by the method specified in ASTM C500, Sections 17 and 18. Type I asbestos-cement pipe has no chemical requirement with regard to uncombined calcium hydroxide. Type II pipe may contain no more than 1 percent calcium hydroxide. Table 4 gives the test results, which are the averages of triplicate tests.

**Table 2**  
**Crushing Strength Tests**

| Specimen<br>Set Number | ASTM<br>Number  | Requirements/1 ft (0.3 m)      | Results/1 ft (0.3 m)                |
|------------------------|-----------------|--------------------------------|-------------------------------------|
| 1                      | —*              | —*                             | greater than 10,000 lb<br>(4545 kg) |
| 2                      | —*              | —*                             | greater than 10,000 lb<br>(4545 kg) |
| 3                      | C296 Sect. 9    | 4000 lb (minimum)<br>(1818 kg) | 9600 lb<br>(4364 kg)                |
| 4                      | C296 Sect. 9    | 4000 lb (minimum)<br>(1818 kg) | 9360 lb<br>(4254 kg)                |
| 5                      | C428 Sect. 7    | 2400 lb (minimum)<br>(1091 kg) | 4380 lb<br>(1991 kg)                |
| 6                      | C248 Sect. 7    | 2400 lb (minimum)<br>(1091 kg) | 5920 lb<br>(2691 kg)                |
| 7                      | C644 Sect. 10.3 | 2400 lb (minimum)<br>(1091 kg) | 3460 lb<br>(1573 kg)                |

\*These pipes were tested with the polyurethane foam insulation and exterior filament wound glass reinforced plastic in place. ASTM does not have a standard test for this configuration.



**Figure 1.** Crushing strength setup.

**Table 3**  
**Sizes and Dimensions**

| Specimen Set Number | ASTM Number     | Requirement   | Results, In.                              | Difference, In.        |
|---------------------|-----------------|---|---|------------------------|
| 1                   | C296 Sect. 12.2 | Not more than 5 percent less than nominal (allowed 0.5) | 9.75/9.80<br>9.62/9.75<br>9.75/9.80       | 0.38/0.20              |
| 2                   | C296 Sect. 12.2 | Not more than 5 percent less than nominal               | 9.75/9.80<br>9.75/9.80<br>9.68/9.87       | 0.32/0.13<br>0.32/0.13 |
| 3                   | C296 Sect. 12.2 | Not more than 5 percent less than nominal (allowed 0.3) | Could not measure due to lining condition |                        |
| 4                   | C296 Sect. 12.2 | Not more than 5 percent less than nominal (allowed 0.4) | 7.75/7.75<br>7.70/7.75<br>7.70/7.75       | 0.30/0.25              |
| 5                   | C428 Sect. 11.2 | Not more than 1/4 in. less than nominal                 | 7.80/7.90<br>7.75/7.80<br>7.85/7.90       | 0.25/0.10              |
| 6                   | C428 Sect. 11.2 | Not more than 1/4 in. less than nominal                 | 7.92/7.92<br>7.87/7.92<br>7.88/8.00       | 0.13/0.0               |
| 7                   | C644 Sect. 7.2  | Not more than 1/4 in. less than nominal                 | 5.93/5.97<br>5.83/5.96<br>5.83/5.94       | 0.17/0.03              |

Metric conversion factor: inches  $\times$  25.4 = mm

**Table 4**  
**Chemical Requirements**

| Specimen Set Number | ASTM Number        | Requirement  | Results       |
|---------------------|--------------------|--|---------------|
| 1                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.162 percent |
| 2                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.220 percent |
| 3                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.226 percent |
| 4                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.493 percent |
| 5                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.346 percent |
| 6                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.132 percent |
| 7                   | C500 Sect. 17 & 18 | Not more than 1 percent uncombined calcium hydroxide | 0.214 percent |

**Summary of Results:** all pipe specimens qualify as Type II with respect to uncombined calcium hydroxide content. However, this does not mean that the in-service pipe (specimen sets 3 and 5) which had been exposed to soil water necessarily met the Type II requirements before such exposure. It is possible that saturation of the pipe with solutions of mineral salts may have led to reaction or the residual calcium hydroxide.

#### Soil Tests

The two soil samples provided were tested to determine soluble sulfate and chloride content. The samples were thoroughly dried at 212°F (100°C) overnight in a circulating air oven. A specimen of the finer components of the soils (gravel was excluded) was weighed out and then combined with an equal weight of deionized water. This mixture was allowed to sit (with occasional stirring) in a closed container for 36 hours. A portion of each supernatant liquid leachate was taken. This leachate was diluted 100:1 with deionized water, and a standard turbidimetric method was used to establish the sulfate ion content.<sup>6</sup> Both soils had soluble sulfate contents of about 15,000 ppm. These results are consistent with the Dames and Moore study which showed values of 9,000 ppm, 12,000 ppm, and 15,000 ppm for three locations in the test area.<sup>7</sup> The leachate pH was about 5, as measured by color change comparison paper. This is inconsistent with the Dames and Moore finding (slightly alkaline) and with the presence of coral and limestone in the area. The leachate's acidity cannot be explained.

The chloride ion content of the soil leachate was measured by a chloride-ion electrode ion meter. The soil sample designated "No. 3" by MED (Rear) had a chloride ion concentration of 41,750 ppm. These results agree with the Dames and Moore study, which found 18,000 ppm to 60,000 ppm.

#### Water Test

The soluble sulfate content of the water sample provided was measured as described above, except

no dilution factor was used. The sulfate ion concentration was 250 ppm. Since the water was from the chilled water system, it appears to have been treated and can be presumed to contain other solubles consistent with a "hard" tap water. No organics were found in the water sample.

#### Lining Tests

Numerous evaluations and studies were performed on the epoxy-lining material\* used in the asbestos-cement pipe. These studies included composition, lining thickness, adhesion to the pipe material under severe conditions, hardness, and stability when exposed to acidic, alkaline, and neutral liquid submersion.

#### Composition

The plastic lining used in the asbestos-cement pipe was made from an epoxy resin of epichlorohydrin reacted with bisphenol A (epoxide equivalent weight of 175 to 210). This resin was cured by mixing with an aliphatic amine compound, either diethylenetriamine (DTA) or triethylenetetramine (TETA). Curing occurs by a series of complex chemical reactions which ultimately result in a three-dimensional, cross-linked polymer network structure. The type and concentration of curing agent, the curing conditions, and other substances present all have some effect on the final polymer structure and morphology, and thus on the polymer's physical and chemical properties.

The epoxy pipe lining under study also contained a colorant and a finely divided particulate silica filler. The purpose of the colorant is not known—although it does allow visual confirmation of the coating. Silica is often used in epoxies to increase their resistance to abrasion.

As described on p. 8, the pipe lining was applied as a liquid and centrifugally cast against the inner surface of the pipe while the curing process proceeded. Final curing was promoted by heating the pipe and was assessed by checking whether the lining met a certain minimum hardness value (Barcol 40).

Since the lining application process is held as proprietary information by asbestos-cement pipe manufacturers, the method of formulating the resin mixture, and its mixing and deposition on the pipe substrate could not be learned. All these factors could have an impact on quality control since a variation in any one or all may cause inhomogeneities within a single pipe, or relatively undetectable differences from one pipe section to another.

<sup>6</sup>Standard Methods for the Examination of Water and Wastewater, 14th ed., (American Public Health Association, American Water Works Association, and the Water Pollution Control Federation, 1975), p 496.

<sup>7</sup>Study of Corrosive Soil Conditions and Need for Cathodic Protection, Various [Middle East] Locations, Delivery Order 0005, Contract DACA 75-76-D-0040 (Dames and Moore, August 1976).

The most critical aspect of potential variation in the properties of the cured epoxy is the chemical balance (stoichiometry) between the number of reaction sites available in the epoxy resin and the number of reactive sites available in the curing agent. It is only through these reactions that the network structure can be built up; a deficiency of either type will cause (or allow) instabilities in solvent contact or development of stresses during thermal exposure. At concentrations above and below the stoichiometric levels, both DTA and TETA can enter into secondary reactions that produce heat distortion temperatures that are lower than optimum.<sup>8</sup> Furthermore, the regularity between network connection points (crosslinks) can change with the curing agent's composition. These changes in distances between crosslinks, or crosslink density, can have profound effects on the swelling behavior of the cured resin.<sup>9</sup> The extent of crosslinking can be measured by a swollen particle method, in which a lower value of crosslink density (completion of reaction) is obtained as a function of swelling.<sup>10</sup>

In summary, improper balance between reactants (or lack of uniformity) may cause composition changes in the final epoxy material that could reduce its performance, especially when it is exposed to heat and solvent simultaneously.

#### Thickness

The pipe lining's thickness was measured by removing a piece of lining from the pipe (cleaning off any adhered pipe cementitious material) and measuring the thickness with a micrometer in accordance with ASTM C541.<sup>11</sup> Specimens representing the thickest and thinnest portions of the lining were intentionally removed and measured. Table 5 presents the thicknesses measured from the pipe specimen sets provided.

Summary of Results: the lining thickness varied considerably both within a single pipe and from pipe to pipe. No pattern of variation could be established

relating lining thickness to whether the pipe had been in service (exposed to abrasive conditions).

#### Adhesion

The adhesive bond of the lining to the asbestos-cement pipe substrate was evaluated by testing in accordance with ASTM C541.<sup>12</sup> This test consisted of a subjective, visual examination of bond strength, peeling, and blistering of small sections of the pipe (with lining attached) soaked in distilled water at 150°F (65.5°C) for 96 hours. A separate set of specimens was soaked at the boiling point for 4 hours.

There was no evidence of blisters or peeling of the lining in either test. A suitable specimen could not be obtained from specimen set number 3 due to severe blistering already present.

In all cases, the bond strength of the test specimens was much lower than control specimens of untested pipe. No quantitative adhesive bond strength could be established for either the control or test specimens, but subjective comparison revealed quite low adherence in the test specimens.

The lining material in several of the test specimens was much softer and more pliable at the elevated temperature. A piece of the lining material alone (completely detached from the pipe) from specimen set number 3 was very rubbery in the boiling water and curled up completely on itself. A similar piece in the 150°F (65.5°C) water bath also curled, but not as quickly or severely as in the boiling water.

#### Wetting

The propensity of an epoxy resin to adhere to a substrate can be judged by determining the wetting action at the interface. A puddle of liquid resin mixture was applied to a flat plate of asbestos-cement material, and the angle of the edge of the pool was estimated. Good wetting was indicated by the ease with which the pool of resin spread, and by the very low angle resulting along the edge. Incompatibility of an adhesive and substrate results in a high angle and resistance to flow in an evaluation of this nature.

The initial adhesion of the epoxy lining and the pipe material was excellent; a bonding deficiency was not shown, based on surface tension of the materials or on wetting action.

<sup>8</sup> Henry Lee and Kris Neville, *Epoxy Resins, Their Applications and Technology* (McGraw-Hill Book Company, Inc., 1957), p 64.

<sup>9</sup> C. A. May and Yoshio Tanaka, *Epoxy Resins: Chemistry and Technology* (Marcel Dekker, Inc., 1973), p 330.

<sup>10</sup> W. G. Potter, *Epoxide Resins* (Springer-Verlag, Inc., 1970), p 89.

<sup>11</sup> ASTM C541, "Linings of Asbestos-Cement Pipe," Part II (ASTM, 1978).

<sup>12</sup> ASTM C541, Part 5.



**Table 5**  
**Lining Thickness**

| Specimen<br>Set Number | ASTM<br>Number | Requirement                                | Results,<br>In. (mm)   |
|------------------------|----------------|--|--|
| 1                      | C 541 Sect. 11 | Not less than<br>12 mils (0.3 mm)<br>thick | 0.027 (0.68 mm)<br>0.035 (0.89 mm)<br>0.043 (1.09 mm)<br>0.059 (1.50 mm) |
| 2                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.034 (0.86 mm)<br>0.036 (0.91 mm)<br>0.058 (1.47 mm)<br>0.067 (1.70 mm) |
| 3                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.041 (1.04 mm)<br>0.051 (1.29 mm)<br>0.054 (1.37 mm)<br>0.064 (1.62 mm) |
| 4                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.037 (0.94 mm)<br>0.042 (1.07 mm)<br>0.043 (1.09 mm)<br>0.051 (1.29 mm) |
| 5                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.036 (0.91 mm)<br>0.038 (0.96 mm)<br>0.080 (2.03 mm)<br>0.089 (2.26 mm) |
| 6                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.029 (0.74 mm)<br>0.040 (1.02 mm)<br>0.044 (1.12 mm)<br>0.052 (1.32 mm) |
| 7                      | C541 Sect. 11  | Not less than<br>12 mils (0.3 mm)<br>thick | 0.015 (0.38 mm)<br>0.016 (0.41 mm)<br>0.019 (0.48 mm)<br>0.025 (0.63 mm) |

\*Produced by a different manufacturer than specimens 1 through 6. The lining material in this pipe did not appear to contain silica filler.

#### Hardness

Shore D durometer hardness measurements were made on the lining material used in the boiling water adhesion tests. ASTM D2240 test procedures were used.<sup>13</sup> The hardness was measured on the lining material before the test (at room temperature) and while the material was conditioned to the boiling water temperature. Table 6 shows the test results.

The important feature shown by the hardness measurement is the range of values obtained at both test temperatures and the obvious differences in the amount of softening that occurred. These variations in hardness and amount of softening suggest a lack of uniform composition of the lining material from pipe to pipe.

<sup>13</sup>ASTM D2240, "Rubber Property—Durometer Hardness," Part 35 (ASTM, 1978).

**Table 6**  
**Hardness Test Results**

| Specimen<br>Set Number | Hardness<br>(Initial) | Hardness<br>(Hot) |
|------------------------|-----------------------|-------------------|
| 1                      | 64                    | 52                |
| 2                      | 80                    | 68                |
| 3                      | 70                    | 60                |
| 4                      | 79                    | 43                |
| 5                      | 80                    | 43                |
| 6                      | 62                    | 46                |
| 7                      | 74                    | 55                |

A second type of hardness test was performed on the lining in specimen set number 3—the lining which displayed very severe deterioration. Rockwell M hardness readings were made on the flow contact surface of the lining, on the bond surface of the lining, and after a few microns of material had been scraped away from each surface.

The Rockwell M hardness of the flow surface was about 95, and of the bond surface about 107. After scraping, these two surfaces gave readings of 86 and 97 respectively.

Similar Rockwell M hardnesses were taken in zones where blistering was obvious. (In this context "blister" refers to a bulge of the entire lining away from the pipe substrate.) Some blisters contained a thin white deposit of material while others did not. The white spots appeared to be caused by water (carrying dissolved material) which filled the blister cavity, and then evaporated, leaving the deposit. The hardness of the lining through the white spot on the bond surface was 115, while the hardness of a similar blister without the white spot was 106. After removal of the white spot deposit by gentle scraping, retesting showed a decrease in hardness to 106.

The Rockwell M hardness tests showed a variation of hardness within a single lining material from surface to surface. On both surfaces, there was a distinct skin effect which was only a few microns thick. The function of the white spot with respect to hardness was probably the result of only slight penetration of the deposited white substance into the lining material.

#### *Infra-Red Analysis*

Detailed analyses were conducted using a Fourier Transform Infra-Red Spectrometer (FTIR). This method allows precise, well-defined spectra to be obtained for study. The degree of resolution is very high, and many individual spectra scans can be stored in the machine and then printed out as an average spectrogram. This spectrogram can then be used to identify the materials present, the degree of crosslinking (by end-group analysis), and numerous other features of the material.

Several hundred spectra were analyzed, and the following major findings were made regarding specimen set 3. This set received the most careful study since it represented the most extensively failed material.

1. Significant differences in epoxy composition existed through the lining thickness, as shown by the presence of more amines near the bond surface. Thus, greater crosslinking of the epoxy resin occurred near the bond face, and this difference could cause greater residual stress near the bond surface.

2. The amount of silica filler was greater at the bond surface. A gradient of silica concentration existed from the flow surface of the lining to the bond surface.

This inhomogeneity in silica dispersion could be the cause of some residual stresses in the cured lining material.

3. The "white spots" (above) are composed of sulfur oxides, primarily sulfates, and some carbonates. These salts were apparently deposited from solution that had permeated the porous asbestos-cement pipe. The sulfate presence in the bond side of the epoxy lining showed only a slight penetration into the lining, with a decreasing amount away from the bond surface. There was essentially no sulfate at or near the flow surface. It appears that the salt solution arrived at the epoxy lining bond interface with the asbestos-cement pipe material by soaking through the pipe rather than through the lining. The salt solution could have adversely affected the adhesive bond, thus allowing other factors, such as residual stress, to separate the lining from the pipe.

#### *Swelling*

Many polymeric materials swell in the presence of solvents. Swelling is related to dissolving. In the case of crosslinked materials, dissolution usually does not occur, so the solvent effect shows up as both weight gain and volume increase. Swollen materials are softer and more pliable. Residual stresses usually cause deformation of the plastic, which relieves the stresses.

The degree or extent of swelling depends on a number of factors, including solvent quality, or effective interaction between solvent and the plastic; molecular weight of the plastic, which in the case of crosslinked materials is taken as the distance between crosslinks; temperature; and internal stresses. Swelling is often reversible, and cyclic exposure to solvent may cause progressive decomposition of the plastic material. Destruction of the network will be more pronounced and rapid if a breakdown of the network (chain scission) occurs in addition to the swelling phenomenon.

Samples of pipe linings were subjected to tests of chemical resistance in accordance with the procedures required by ASTM C541 and ASTM D543.<sup>14</sup> Room temperature was used throughout the test since elevated temperatures may cause effects which do not reflect the durability or resistance of the material and which cannot be directly and confidently related to the results obtained at a lower temperature. Section

<sup>14</sup> ASTM C541, "Linings for Asbestos-Cement Pipe," Part 16 (ASTM, 1978); ASTM D543, "Resistance of Plastics to Chemical Reagents," Part 35 (ASTM, 1979).

9.3 of ASTM C541 and Section 7 of ASTM D543 were used; the exposure media were as described in these sections.

The effects of the four "solvents" or swelling agents can be seen in Tables 7 through 10. The lining material showed different amounts of resistance to the four reagents used, and the results agreed well with Potter.<sup>15</sup> Swelling and weight gain are time dependent, as indicated by the 7- and 30-day results. Acidic media cause much more swelling than does a neutral or an alkaline medium. Acetic acid had a very pronounced effect on some lining materials. Table 11 shows the volume change associated with exposure to the test media.

Note the large degrees of swelling in some cases and the relatively small amount of swelling in others—

<sup>15</sup>W. G. Potter, *Epoxide Resins* (Springer-Verlag, Inc., 1970), p 104.

all within the same medium. The variation of swelling from specimen set to specimen set shows the drastic differences in the resistance of the lining material from each set to the same exposure conditions. Such variations in swelling behavior can help explain why some pipe sections experienced lining failures while others did not.

Additional tests were performed to try to relate crosslinking to the extent of swelling in the test media. Specimens of epoxy resin were cured below and above the stoichiometric requirements. The manufacturers of epoxy resins of the type used for lining materials recommend a range of curing agent proportions that generally spans both sides of the stoichiometric balance point. The test specimens prepared extended well beyond the range recommended so that the effect of serious nonstoichiometric mixing might be detected. Tables 12 through 15 give the results of this study. Note that both aliphatic amine curing agents typically used were included in the study—i.e., DTA and TETA.

Table 7  
Distilled Water Effects on Lining Material

| Specimen Set Number | 1      | 2      | 3      | 4      | 5      | 6      | 7      |
|---------------------|--------|--------|--------|--------|--------|--------|--------|
| Length (in.)        | 2.976  | 3.025  | 3.019  | 2.997  | 2.991  | 2.990  | 3.039  |
| Width (in.)         | 1.051  | 1.092  | 1.079  | 1.090  | 1.129  | 1.048  | 1.159  |
| Thickness (in.)     | 0.046  | 0.045  | 0.049  | 0.056  | 0.037  | 0.039  | 0.013  |
| Volume (cu in.)     | 0.144  | 0.147  | 0.160  | 0.175  | 0.125  | 0.122  | 0.046  |
| Start Weight (g)    | 3.2471 | 3.3932 | 3.5457 | 3.8079 | 2.6482 | 2.6856 | 1.6706 |
| 7-Day Weight (g)    | 3.2608 | 3.4120 | 3.6964 | 3.8248 | 2.9294 | 2.7022 | 1.6839 |
| Δ Weight (g)        | 0.0137 | 0.0188 | 0.1507 | 0.0169 | 0.2812 | 0.0166 | 0.0133 |
| Change (percent)    | 0.42   | 0.55   | 4.25   | 0.44   | 10.62  | 0.62   | 0.80   |
| 30-Day Weight (g)   | 3.2733 | 3.4323 | 3.7206 | 3.8428 | 2.9222 | 2.7148 | 1.6875 |
| Δ Weight (g)        | 0.0262 | 0.0391 | 0.1749 | 0.0349 | 0.2740 | 0.0292 | 0.0169 |
| Change (percent)    | 0.81   | 1.15   | 4.93   | 0.92   | 10.35  | 1.09   | 1.01   |

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

**Table 8**  
**Sulfuric Acid (10 percent) Effects on Lining Material**

| Specimen Set Number | 1       | 2      | 3      | 4      | 5      | 6      | 7      |
|---------------------|---------|--------|--------|--------|--------|--------|--------|
| Length (in.)        | 3.012   | 3.015  | 3.002  | 3.006  | 3.019  | 2.987  | 2.985  |
| Width (in.)         | 1.034   | 1.101  | 1.065  | 1.117  | 1.126  | 1.099  | 1.073  |
| Thickness (in.)     | 0.045   | 0.046  | 0.052  | 0.054  | 0.035  | 0.038  | 0.016  |
| Volume (cu in.)     | 0.140   | 0.153  | 0.166  | 0.181  | 0.119  | 0.125  | 0.051  |
| Start Weight (g)    | 3.1066  | 3.3202 | 3.5704 | 4.0753 | 2.5124 | 2.8556 | 1.1218 |
| 7-Day Weight (g)    | 3.0794  | 3.8318 | 5.1372 | 4.1881 | 4.0128 | 3.1444 | 1.1535 |
| Δ Weight (g)        | -0.0272 | 0.5116 | 1.5668 | 0.1126 | 1.5004 | 0.2888 | 0.0317 |
| Change (percent)    | -0.88   | 15.41  | 43.88  | 2.76   | 59.72  | 10.11  | 2.83   |
| 30-Day Weight (g)   | 3.4053  | 3.8188 | 5.1220 | 4.3535 | 4.5585 | 3.1928 | 1.1940 |
| Δ Weight (g)        | 0.2987  | 0.4985 | 1.5516 | 0.2780 | 2.0461 | 0.3372 | 0.0722 |
| Change (percent)    | 4.62    | 15.01  | 43.46  | 6.82   | 81.44  | 11.81  | 6.44   |

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

**Table 9**  
**Acetic Acid (5 percent) Effects on Lining Material**

| Specimen Set Number | 1      | 2      | 3      | 4      | 5      | 6      | 7      |
|---------------------|--------|--------|--------|--------|--------|--------|--------|
| Length (in.)        | 3.003  | 3.015  | 3.032  | 2.990  | 3.011  | 3.046  | 3.032  |
| Width (in.)         | 1.056  | 1.076  | 1.058  | 1.079  | 1.131  | 1.088  | 1.075  |
| Thickness (in.)     | 0.045  | 0.046  | 0.053  | 0.044  | 0.035  | 0.090  | 0.015  |
| Volume (cu in.)     | 0.143  | 0.149  | 0.170  | 0.142  | 0.119  | 0.133  | 0.049  |
| Start Weight (g)    | 3.3012 | 3.2533 | 3.6494 | 3.2157 | 2.5176 | 2.9119 | 1.0779 |
| 7-Day Weight (g)    | 3.3239 | 4.6867 | *      | 3.2685 | *      | 3.0801 | 1.0960 |
| Δ Weight (g)        | 0.0227 | 1.4334 | *      | 0.0528 | *      | 0.1682 | 0.0181 |
| Change (percent)    | 0.69   | 44.06  | *      | 1.64   | *      | 5.78   | 1.68   |
| 30-Day Weight (g)   | 3.3498 | 4.6954 | *      | 3.3519 | *      | 3.3289 | 1.1117 |
| Δ Weight (g)        | 0.0486 | 1.4421 | *      | 0.1362 | *      | 0.4170 | 0.0338 |
| Change (percent)    | 1.4/   | 44.33  | *      | 4.25   | *      | 14.32  | 3.14   |

\*Sample disintegrated. Residue on bottom of vessel ranged in size from particles around 1/1000 in. (0.025 mm) in diameter to 1 in.-long (25.4 mm) strips, 1/4 in. (6.35 mm) wide.

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

**Table 10**  
**Sodium Hydroxide (5 percent) Effects on Lining Material**

| Specimen Set Number | 1      | 2      | 3      | 4      | 5      | 6      | 7      |
|---------------------|--------|--------|--------|--------|--------|--------|--------|
| Length (in.)        | 3.007  | 2.946  | 3.018  | 2.940  | 3.004  | 3.006  | 3.026  |
| Width (in.)         | 1.040  | 1.075  | 1.074  | 1.117  | 1.127  | 1.106  | 1.088  |
| Thickness (in.)     | 0.043  | 0.043  | 0.050  | 0.051  | 0.034  | 0.039  | 0.013  |
| Volume (cu in.)     | 0.134  | 0.136  | 0.162  | 0.167  | 0.115  | 0.130  | 0.043  |
| Start Weight (g)    | 3.0986 | 3.0603 | 3.6061 | 3.7914 | 2.5051 | 2.8035 | 1.0062 |
| 7-Day Weight (g)    | 3.1095 | 3.0764 | 3.7619 | 3.8064 | 2.7136 | 2.8217 | 1.0152 |
| Δ Weight (g)        | 0.0109 | 0.0161 | 0.1558 | 0.0150 | 0.2085 | 0.0182 | 0.0090 |
| Change (percent)    | 0.35   | 0.53   | 4.32   | 0.40   | 8.32   | 0.65   | 0.89   |
| 30-Day Weight (g)   | 3.1164 | 3.0919 | 3.8639 | 3.8244 | 2.8340 | 2.8320 | 1.0156 |
| Δ Weight (g)        | 0.0178 | 0.0316 | 0.2578 | 0.0330 | 0.3289 | 0.0285 | 0.0094 |
| Change (percent)    | 0.57   | 1.03   | 7.15   | 0.87   | 13.13  | 1.02   | 0.93   |

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

**Table 11**  
**Swelling of Lining Material**

| Medium                | Specimen Set Number | Length (in.)      | Width (in.) | Thickness (in.) | Volume (cu in.) | Δ Volume (cu in.) | Change in Volume (%) |
|-----------------------|---------------------|-------------------|-------------|-----------------|-----------------|-------------------|----------------------|
| Water                 | 1                   | 2.734             | 1.054       | 0.046           | 0.145           | 0.001             | 0.69                 |
|                       | 2                   | 3.023             | 1.101       | 0.045           | 0.150           | 0.001             | 0.67                 |
|                       | 3                   | 3.046             | 1.087       | 0.051           | 0.169           | 0.009             | 5.63                 |
|                       | 4                   | 3.005             | 1.127       | 0.052           | 0.176           | 0.001             | 0.57                 |
|                       | 5                   | 3.076             | 1.160       | 0.038           | 0.136           | 0.011             | 8.80                 |
|                       | 6                   | 3.010             | 1.108       | 0.038           | 0.126           | 0.001             | 0.80                 |
|                       | 7                   | 3.046             | 1.469       | 0.013           | 0.146           | 0.010             | 7.14                 |
| Sulfuric Acid (10%)   | 1                   | 3.022             | 1.131       | 0.044           | 0.150           | 0.010             | 7.14                 |
|                       | 2                   | 3.235             | 1.209       | 0.041           | 0.160           | 0.007             | 4.58                 |
|                       | 3                   | 3.181             | 1.127       | 0.062           | 0.222           | 0.056             | 33.73                |
|                       | 4                   | 3.107             | 1.126       | 0.056           | 0.196           | 0.015             | 8.29                 |
|                       | 5                   | 3.171             | 1.150       | 0.052           | 0.190           | 0.071             | 59.66                |
|                       | 6                   | 3.020             | 1.036       | 0.045           | 0.141           | 0.016             | 12.80                |
|                       | 7                   | 2.82              | 1.098       | 0.016           | 0.053           | 0.002             | 3.92                 |
| Acetic Acid (5%)      | 1                   | 3.016             | 1.057       | 0.046           | 0.147           | 0.004             | 2.80                 |
|                       | 2                   | 3.413             | 1.221       | 0.050           | 0.208           | 0.059             | 39.60                |
|                       | 3                   | INFINITE SWELLING |             |                 |                 |                   |                      |
|                       | 4                   | 3.005             | 1.080       | 0.045           | 0.146           | 0.004             | 2.82                 |
|                       | 5                   | INFINITE SWELLING |             |                 |                 |                   |                      |
|                       | 6                   | 3.083             | 1.098       | 0.045           | 0.152           | 0.019             | 14.29                |
|                       | 7                   | 3.025             | 1.074       | 0.015           | 0.049           | 0.000             | 0.00                 |
| Sodium Hydroxide (5%) | 1                   | 3.008             | 1.042       | 0.044           | 0.138           | 0.004             | 2.99                 |
|                       | 2                   | 2.946             | 1.079       | 0.043           | 0.137           | 0.001             | 0.74                 |
|                       | 3                   | 3.041             | 1.081       | 0.052           | 0.171           | 0.009             | 5.56                 |
|                       | 4                   | 2.962             | 1.120       | 0.051           | 0.169           | 0.002             | 1.20                 |
|                       | 5                   | 3.082             | 1.159       | 0.036           | 0.128           | 0.013             | 11.30                |
|                       | 6                   | 3.011             | 1.103       | 0.039           | 0.130           | 0.000             | 0.00                 |
|                       | 7                   | 3.025             | 1.100       | 0.013           | 0.049           | 0.000             | 0.00                 |

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

**Table 12**  
**Effect of Curing Agent Concentration on Swelling Behavior in Distilled Water**

| Specimen<br>Set Number      | 1      | 2      | 3      | 4*     | 5*     | 6*     | 7      | 8      | 9      | 10     | 11     | 12*    | 13*    | 14*    | 15     | 16     |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Curing Agent<br>(C.A.)      | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   |
| Wt. C.A.<br>(g/100 g resin) | 5.0    | 6      | 7      | 8      | 10     | 12     | 14     | 15     | 7      | 8      | 9      | 10     | 12     | 14     | 15     | 16     |
| Length (in.)                | 3.012  | 3.056  | 3.054  | 3.052  | 3.038  | 2.987  | 3.029  | 3.055  | 3.038  | 3.037  | 3.072  | 3.028  | 2.997  | 2.982  | 3.027  | 3.023  |
| Width (in.)                 | 1.092  | 1.065  | 1.121  | 1.088  | 1.107  | 1.117  | 1.090  | 1.118  | 1.112  | 1.100  | 1.132  | 1.098  | 1.130  | 1.063  | 1.083  | 1.056  |
| Thickness (in.)             | 0.044  | 0.035  | 0.035  | 0.058  | 0.066  | 0.032  | 0.011  | 0.041  | 0.030  | 0.043  | 0.026  | 0.016  | 0.027  | 0.044  | 0.021  | 0.110  |
| Volume (cu in.)             | 0.145  | 0.114  | 0.121  | 0.193  | 0.222  | 0.107  | 0.036  | 0.140  | 0.101  | 0.144  | 0.090  | 0.053  | 0.078  | 0.139  | 0.069  | 0.351  |
| Start Wt. (g)               | 2.3818 | 2.1799 | 2.1468 | 3.3141 | 4.0616 | 1.9961 | 0.7088 | 1.9490 | 1.9582 | 1.9608 | 2.3061 | 1.0054 | 1.3121 | 2.6717 | 1.0464 | 5.2803 |
| 7-Day Wt. (g)               | 2.4004 | 2.1983 | 2.1660 | 3.3418 | 4.0901 | 2.0143 | 0.7905 | 1.9916 | 1.9779 | 1.9898 | 2.3291 | 1.0191 | 1.3340 | 2.7044 | 1.0672 | 5.3086 |
| Δ Wt. (g)                   | 0.0186 | 0.0184 | 0.0192 | 0.0227 | 0.0285 | 0.0182 | 0.0117 | 0.0426 | 0.0197 | 0.0290 | 0.0230 | 0.0137 | 0.0219 | 0.0327 | 0.0208 | 0.0283 |
| Change (%)                  | 0.78   | 0.84   | 0.84   | 0.84   | 0.70   | 0.91   | 1.50   | 2.19   | 1.01   | 1.48   | 1.00   | 1.36   | 1.67   | 1.22   | 1.99   | 0.54   |
| 30-Day Wt. (g)              | 2.4183 | 2.2098 | 2.1771 | 3.3599 | 4.1230 | 2.0298 | 0.2928 | 2.0110 | 1.9886 | 2.0006 | 2.3427 | 1.0211 | 1.3392 | 2.7323 | 1.0708 | 5.3450 |
| Δ Wt. (g)                   | 0.0365 | 0.0299 | 0.0303 | 0.0408 | 0.0614 | 0.0337 | 0.0140 | 0.0620 | 0.0304 | 0.0398 | 0.0366 | 0.0157 | 0.0271 | 0.0606 | 0.244  | 0.0647 |
| Change (%)                  | 1.53   | 1.37   | 1.41   | 1.23   | 1.51   | 1.69   | 1.80   | 3.13   | 1.55   | 2.03   | 1.59   | 1.56   | 2.07   | 2.27   | 2.33   | 1.23   |

\*Data in these columns are within product specifications; data in other columns are not.

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

Table 13  
Effect of Curing Agent Concentration on Swelling Behavior in 10 Percent Sulfuric Acid

| Specimen<br>Set Number      | 1      | 2      | 3      | 4*     | 5*     | 6*     | 7      | 8      | 9      | 10     | 11     | 12*    | 13*    | 14*    | 15     | 16     |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Curing Agent<br>(C.A.)      | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   |
| Wt. C.A.<br>(g/100 g resin) | 5      | 6      | 7      | 8      | 10     | 12     | 14     | 15     | 7      | 8      | 9      | 10     | 12     | 14     | 15     | 16     |
| Length (in.)                | 2.921  | 3.076  | 3.074  | 3.040  | 3.047  | 2.945  | 2.946  | 3.017  | 3.029  | 3.002  | 3.071  | 3.071  | 3.018  | 2.998  | 2.942  | 3.000  |
| Width (in.)                 | 1.119  | 1.127  | 1.096  | 1.123  | 1.141  | 1.102  | 1.075  | 1.100  | 1.068  | 1.107  | 1.125  | 1.128  | 1.080  | 1.047  | 1.065  | 1.035  |
| Thickness (in.)             | 0.092  | 0.037  | 0.068  | 0.042  | 0.077  | 0.080  | 0.046  | 0.028  | 0.033  | 0.018  | 0.053  | 0.025  | 0.018  | 0.055  | 0.073  | 0.111  |
| Volume (cu in.)             | 0.301  | 0.128  | 0.229  | 0.143  | 0.268  | 0.260  | 0.146  | 0.093  | 0.109  | 0.060  | 0.183  | 0.087  | 0.059  | 0.173  | 0.229  | 0.345  |
| Start Wt. (g)               | 5.1212 | 2.1937 | 4.1103 | 3.2395 | 4.7649 | 4.4452 | 2.7817 | 1.4652 | 2.0243 | 1.0963 | 3.1805 | 1.6044 | 1.0858 | 3.0528 | 3.7874 | 5.9473 |
| 7-Day Wt. (g)               | 5.1446 | 2.2136 | 4.1377 | 2.2700 | 4.8151 | 4.5301 | 2.9085 | 1.6095 | 2.0488 | 1.1502 | 3.2287 | 1.6428 | 1.1351 | 3.1417 | 3.8861 | 6.0571 |
| Δ Wt. (g)                   | 0.0234 | 0.0119 | 0.0274 | 0.0305 | 0.0502 | 0.0849 | 0.1268 | 0.1443 | 0.0245 | 0.0539 | 0.0482 | 0.0384 | 0.0493 | 0.0889 | 0.0987 | 0.1098 |
| Change (%)                  | 0.46   | 0.91   | 0.67   | 1.36   | 1.05   | 1.91   | 4.56   | 9.85   | 1.21   | 4.92   | 1.52   | 2.39   | 4.54   | 2.91   | 2.61   | 1.85   |
| 30-Day Wt. (g)              | 5.1696 | 2.2223 | 4.1637 | 2.2869 | 4.8585 | 4.6282 | 3.0678 | 1.7419 | 2.0880 | 1.1663 | 3.2504 | 1.6511 | 1.1486 | 3.1991 | 3.9706 | 6.1854 |
| Δ Wt. (g)                   | 0.0484 | 0.0286 | 0.0534 | 0.0474 | 0.0936 | 0.1830 | 0.2861 | 0.2767 | 0.0337 | 0.0700 | 0.0699 | 0.0467 | 0.0628 | 0.1463 | 0.1832 | 0.2381 |
| Change (%)                  | 0.95   | 1.30   | 1.30   | 2.12   | 1.96   | 4.12   | 10.29  | 18.88  | 1.66   | 6.39   | 2.20   | 2.91   | 5.78   | 4.79   | 4.84   | 4.00   |

\*Data in these columns are within product specifications; data in other columns are not.

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

Table 14  
Effect of Curing Agent Concentration on Swelling Behavior in 5 Percent Acetic Acid

| Specimen<br>Set Number      | 1      | 2      | 3      | 4*     | 5*     | 6*     | 7      | 8      | 9      | 10     | 11     | 12*    | 13*    | 14*    | 15     | 16     |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Curing Agent<br>(C.A.)      | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   |
| Wt. C.A.<br>(g/100 g resin) | 5      | 6      | 7      | 8      | 10     | 12     | 14     | 15     | 7      | 8      | 9      | 10     | 12     | 14     | 15     | 16     |
| Length (in.)                | 2.938  | 3.042  | 3.028  | 3.025  | 3.038  | 3.050  | 3.027  | 2.920  | 3.058  | 3.036  | 3.100  | 2.995  | 3.039  | 3.035  | 2.929  | 2.955  |
| Width (in.)                 | 1.093  | 1.088  | 1.113  | 1.100  | 1.132  | 1.113  | 1.101  | 1.090  | 1.118  | 1.088  | 1.131  | 1.055  | 1.089  | 1.034  | 1.056  | 1.060  |
| Thickness (in.)             | 0.088  | 0.058  | 0.070  | 0.071  | 0.079  | 0.075  | 0.058  | 0.087  | 0.043  | 0.032  | 0.065  | 0.069  | 0.029  | 0.054  | 0.095  | 0.120  |
| Volume (cu in.)             | 0.283  | 0.192  | 0.236  | 0.236  | 0.272  | 0.255  | 0.913  | .277   | 0.147  | 0.106  | 0.228  | 0.218  | 0.096  | 0.169  | 0.294  | 0.376  |
| Start Wt. (g)               | 4.6739 | 2.9769 | 4.1103 | 3.7755 | 4.6119 | 4.3708 | 3.5789 | 3.9819 | 2.6319 | 1.5727 | 4.0214 | 4.0523 | 1.7541 | 2.8526 | 5.4091 | 6.4364 |
| 7-Day Wt. (g)               | 4.7005 | 2.9992 | 4.1371 | 3.8071 | 4.6521 | 4.4361 | 3.6855 | 4.1388 | 2.6561 | 1.6409 | 4.0595 | 4.0980 | 1.8064 | 2.9167 | 5.5004 | 6.5255 |
| Δ Wt. (g)                   | 0.0266 | 0.0223 | 0.0268 | 0.0316 | 0.0402 | 0.0653 | 0.1066 | 0.1569 | 0.0242 | 0.0682 | 0.0381 | 0.0457 | 0.0523 | 0.0741 | 0.0913 | 0.0891 |
| Change (%)                  | 0.57   | 0.75   | 0.65   | 0.84   | 0.87   | 1.49   | 2.98   | 3.94   | 0.92   | 4.34   | 0.95   | 1.13   | 2.98   | 2.60   | 1.69   | 1.38   |
| 30-Day Wt. (g)              | 4.7443 | 3.0190 | 4.1646 | 3.8405 | 4.6897 | 4.4906 | 3.7876 | 4.2755 | 2.6801 | 1.6370 | 4.0915 | 4.1179 | 1.8197 | 2.9620 | 5.5573 | 6.6158 |
| Δ Wt. (g)                   | 0.0704 | 0.0428 | 0.0543 | 0.0650 | 0.0778 | 0.1198 | 0.2087 | 0.2936 | 0.0482 | 0.0643 | 0.0701 | 0.0656 | 0.0656 | 0.1094 | 0.1482 | 0.1794 |
| Change (%)                  | 1.51   | 1.41   | 1.32   | 1.72   | 1.69   | 2.74   | 5.83   | 7.37   | 1.83   | 4.09   | 1.74   | 1.62   | 3.74   | 3.84   | 2.74   | 2.79   |

\*Data in these columns are within product specifications; data in other columns are not.

Conversion factors: inches x 25.4 = mm  
cubic inches x 1.6 = cm<sup>3</sup>



**Table 15**  
**Effect of Curing Agent Concentration on Swelling Behavior in 5 Percent Sodium Hydroxide**

| Specimen<br>Set Number      | 1      | 2      | 3      | 4*     | 5*     | 6*     | 7      | 8      | 9      | 10     | 11     | 12*    | 13*    | 14*    | 15     | 16     |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Curing Agent<br>(C.A.)      | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | DTA    | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   | TETA   |
| Wt. C.A.<br>(g/100 g resin) | 5      | 6      | 7      | 8      | 10     | 12     | 14     | 15     | 7      | 8      | 9      | 10     | 12     | 14     | 15     | 16     |
| Length (in.)                | 3.004  | 3.062  | 3.084  | 3.053  | 3.036  | 3.057  | 2.979  | 3.013  | 3.069  | 3.022  | 3.016  | 3.023  | 3.060  | 2.949  | 2.987  | 3.025  |
| Width (in.)                 | 1.122  | 1.110  | 1.095  | 1.106  | 1.133  | 1.042  | 1.056  | 1.079  | 1.125  | 1.091  | 1.134  | 1.100  | 1.100  | 1.055  | 1.060  | 1.067  |
| Volume (cu in.)             | 0.155  | 0.105  | 0.132  | 0.101  | 0.268  | 0.160  | 0.170  | 0.140  | 0.145  | 0.102  | 0.116  | 0.166  | 0.158  | 0.134  | 0.266  | 0.336  |
| Start Wt. (g)               | 2.6546 | 1.7378 | 2.3497 | 2.0250 | 4.6240 | 2.8852 | 3.3080 | 2.1074 | 2.6926 | 1.7660 | 2.3519 | 2.6891 | 3.0372 | 2.4159 | 4.3662 | 4.9797 |
| 7-Day Wt. (g)               | 2.6768 | 1.7537 | 2.3689 | 2.0432 | 4.6506 | 2.9143 | 3.3476 | 2.1525 | 2.7154 | 1.7922 | 2.3757 | 2.7140 | 3.0708 | 2.4478 | 4.4051 | 5.0131 |
| Δ Wt. (g)                   | 0.0222 | 0.0159 | 0.0192 | 0.0182 | 0.0266 | 0.0291 | 0.0396 | 0.0451 | 0.0228 | 0.0262 | 0.0238 | 0.0249 | 0.0336 | 0.0319 | 0.0389 | 0.0334 |
| Change (%)                  | 0.84   | 0.91   | 0.82   | 0.90   | 0.58   | 1.01   | 1.20   | 2.14   | 0.85   | 1.48   | 1.01   | 0.93   | 1.11   | 1.32   | 0.89   | 0.67   |
| 30-Day Wt. (g)              | 2.6858 | 1.7596 | 2.3787 | 2.0521 | 4.6881 | 2.4410 | 3.3787 | 2.1765 | 2.7284 | 1.8052 | 2.3836 | 2.7263 | 3.0899 | 2.4737 | 4.4415 | 5.0666 |
| Δ Wt. (g)                   | 0.0312 | 0.0218 | 0.0290 | 0.0271 | 0.0591 | 0.0558 | 0.0707 | 0.0691 | 0.0358 | 0.0392 | 0.0317 | 0.0372 | 0.0529 | 0.0578 | 0.0753 | 0.0896 |
| Change (%)                  | 1.18   | 1.25   | 1.23   | 1.34   | 1.28   | 1.93   | 2.14   | 3.28   | 1.33   | 2.22   | 1.35   | 1.38   | 1.74   | 2.39   | 1.72   | 1.75   |

\*Data in these columns are within product specifications; data in other columns are not.

Conversion factors: inches × 25.4 = mm  
cubic inches × 1.6 = cm<sup>3</sup>

The relationship between weight change and volume change was seen as being reasonably near the same at swellings up to 30 percent (Figure 2); since weight change was much easier to measure, weight gain was used in graphing the results from this series of tests. In the DTA-cured samples there was generally a continuous increase in weight gains as the DTA concentration was increased (Figure 3). The same trend was not present in the TETA-cured materials (Figure 4). The erratic swelling of the TETA-cured materials has not been satisfactorily explained.

It is clear from these studies that off-proportion mixing of epoxy resin and curing agent can cause large amounts of swelling. If the materials for lining each section of pipe are mixed by batch, or if a machine mixer is used and is turned off between lining application in each section of pipe, the concentration of curing agent in the lining may vary. Even relatively

small variations in curing agent can cause large changes in swelling, which both weaken the lining material and cause dimensional changes that could lead to failure.

Consider the swelling of lining material in specimen sets 3 and 5. The large change in dimensions (Table 11) in acidic media would cause the lining to tend either to detach completely from the pipe or to form a number of blisters to compensate for the additional length due to swelling. The extent of blistering would thus relate directly to the amount of swelling. Furthermore, once blisters formed, they would be quite vulnerable to the turbulent effects of material flow within the pipe and, due to the weakened condition of the lining material, could easily rupture.

Summary of results: most of the pipe lining materials failed to meet the chemical resistance require-

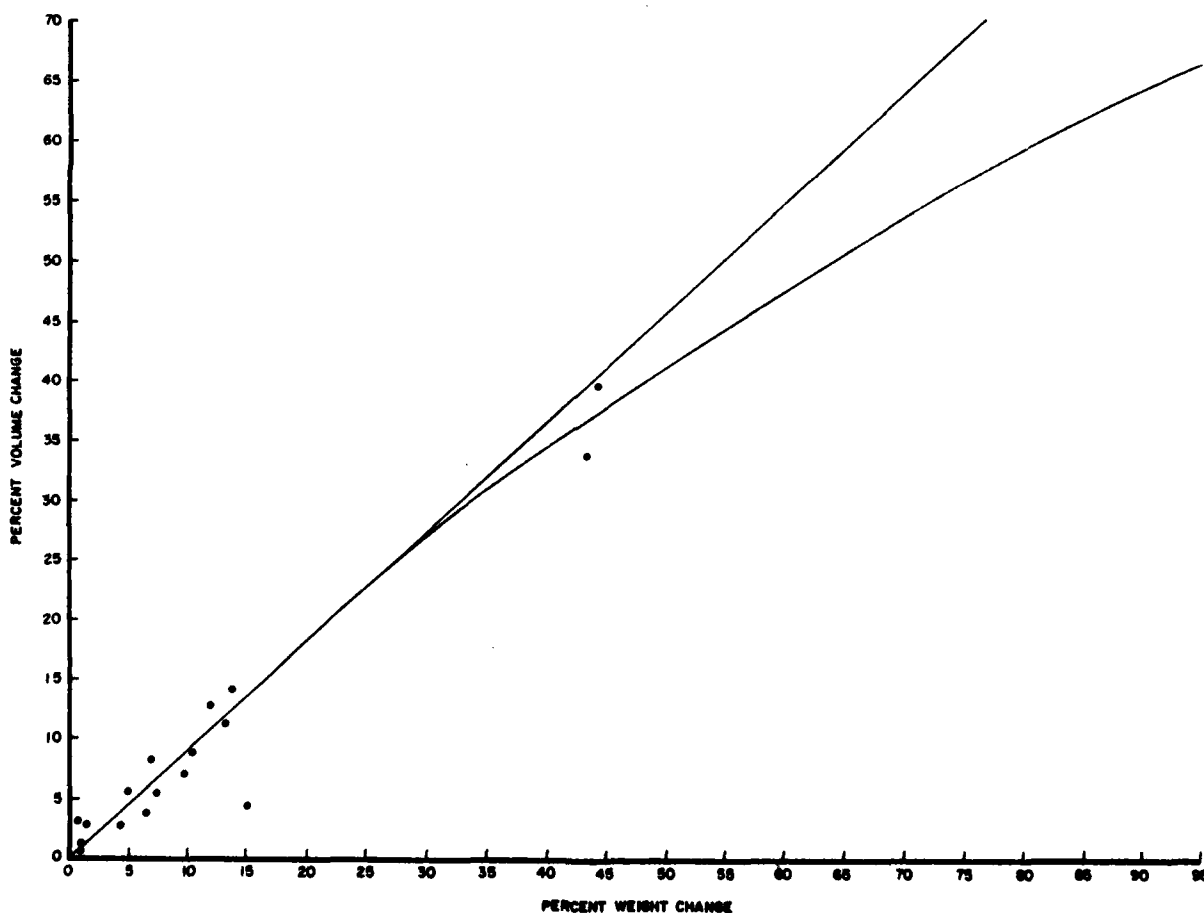


Figure 2. Percent weight change versus percent volume change of epoxy lining material.

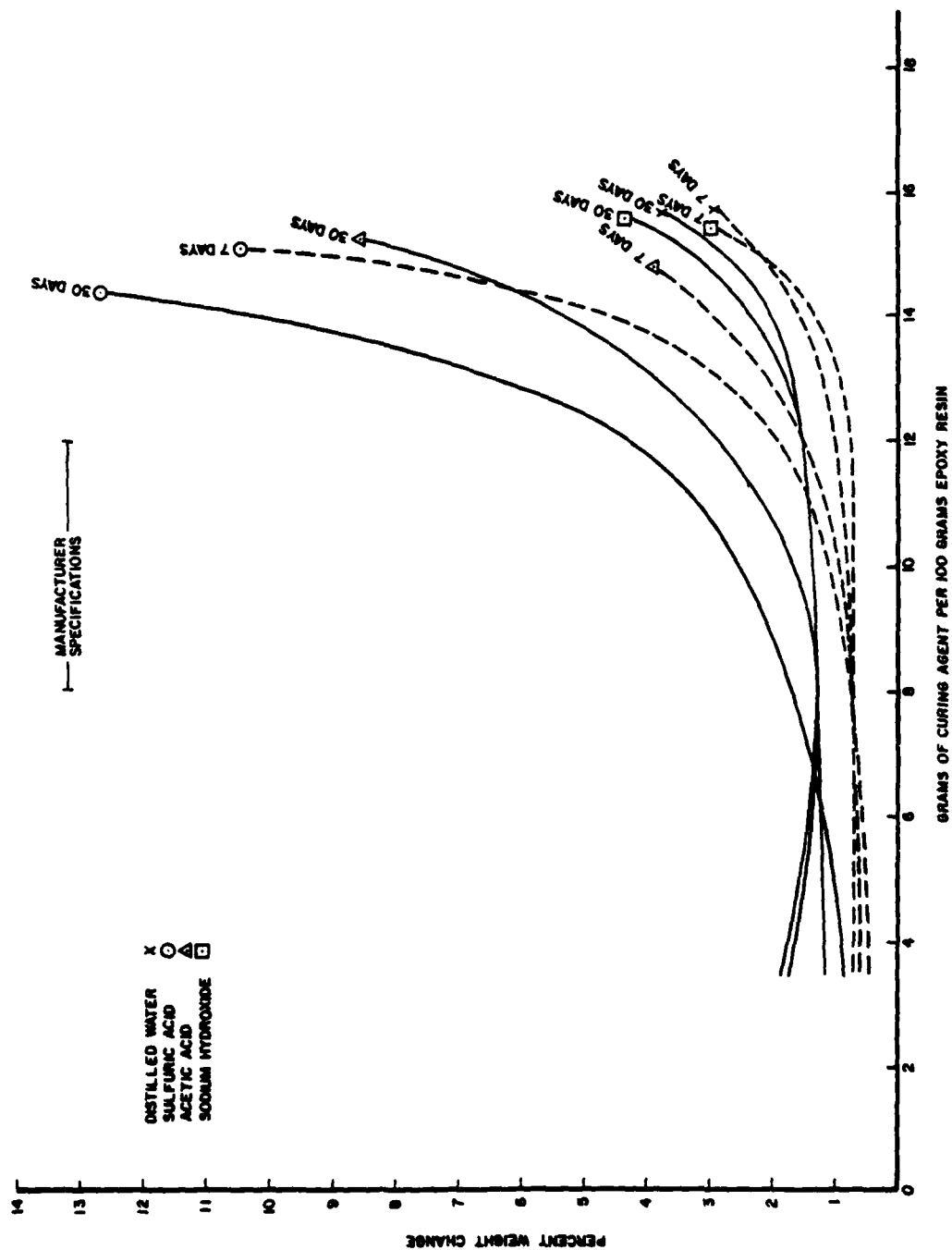


Figure 3. Weight gain versus curing agent (DTA) concentration.

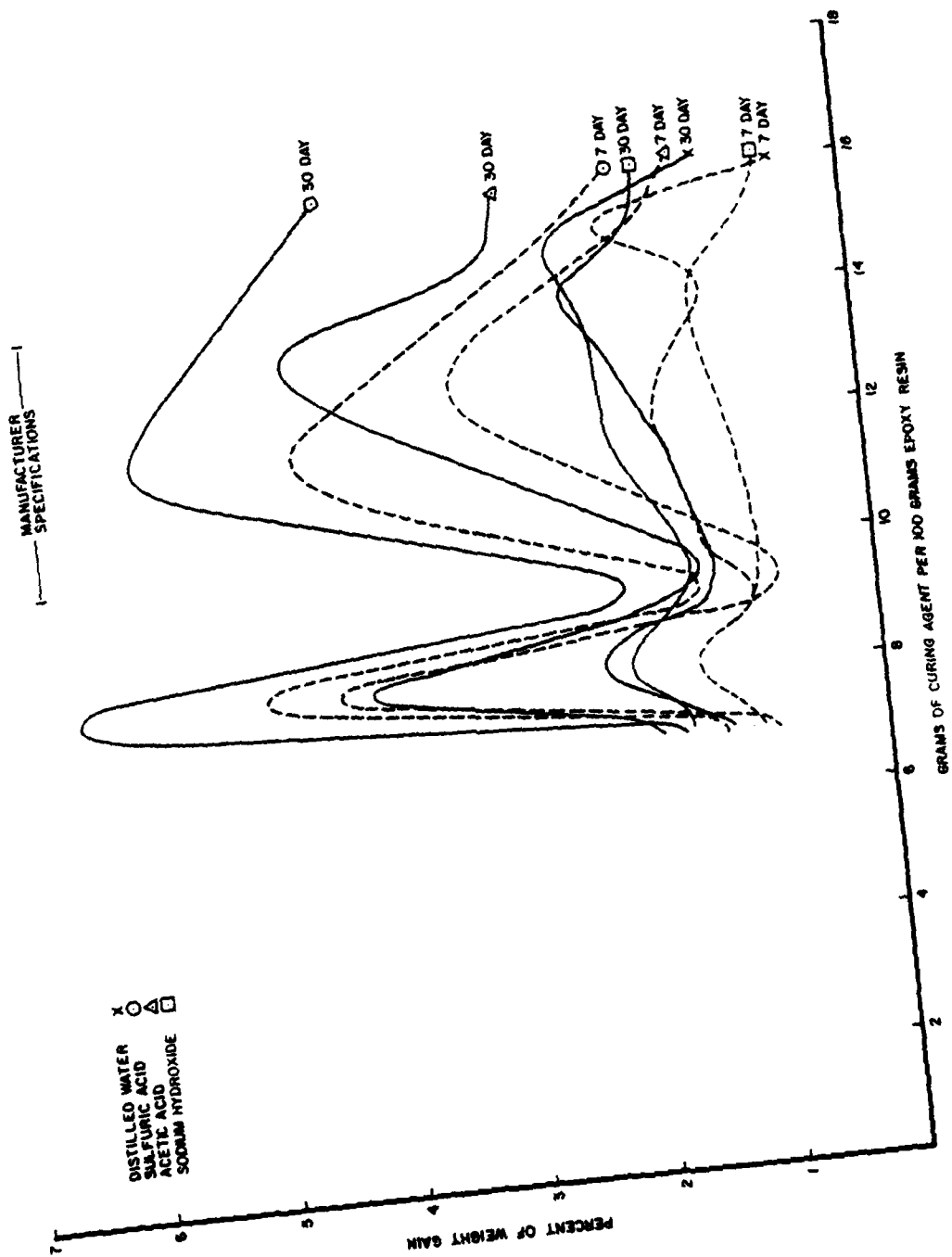


Figure 4. Weight gain versus curing agent (TETA) concentration.

ments of not more than 0.5 percent weight gain after 7 days, or more than 0.9 percent weight gain after 30 days. Off-proportion curing agent was the probable cause of excessive weight gain.

#### *Other Observations*

Visual observations made during the course of the study can also help explain the cause of lining failure.

Residual stresses in the lining material were one of the major findings. When crushing tests were done on specimen set number 3, a section of the lining completely detached from the broken pipe. This piece of lining was about half the circumference of the pipe and the full length of the pipe. Since it was detached there was no resistance to deformation; it was noticed that after less than a week of sitting undisturbed at room temperature the lining had completely inverted its shape. The surface that had been concave (flow surface) was then almost identically curved into a convex shape (Figure 5). This amount of stress relief deformation is very unusual and represents a high degree of potential energy storage. The fact that the stress was relieved at room temperature was also significant since this is well below the glass transition point ( $T_g$  is almost  $100^\circ\text{C}$  for this material). Sources of the residual stress may be summarized as follows:

1. Variation in thickness, crosslinking, and filler content through the thickness undoubtedly caused some of the internal stress.



Figure 5. Change in shape of lining.

2. Centrifugal casting of the lining also helped cause residual stresses, although these are unavoidable in the lining process.

3. Changes in curing agent content and curing schedule from pipe section to pipe section also caused different amounts of residual stress.

Discoloration of the lining material in acidic media was also observed. While this does not indicate any strong adverse effects, a variation of discoloration within a single test coupon does indicate both a non-homogenous mixture when the lining was applied and poor workmanship or quality control on the part of the manufacturer. Figure 6 shows striations of discoloration that occurred in a test coupon from specimen set number 3, the most severely failed pipe provided for study.

Small blisters within the lining material were seen in specimen set number 3. These small blisters were within the lining material, completely enclosed, and all near the flow surface of the lining. They appeared to be the result of trapped air at the time of manufacture. No additional blisters of this type were observed in any of the tests. The blisters ranged from about 1 mm to about 5 mm in diameter and were generally circular. They were quite extensive, as can be seen in Figure 7.

The final visual observation was of the appearance of the lining's flow surface. Some pipe specimen liners were highly glossy, while others were dull, having the appearance of almost a matte finish. Neither condition really suggests a fault with the material, but the difference points out the apparent changes that were allowed in the application process.

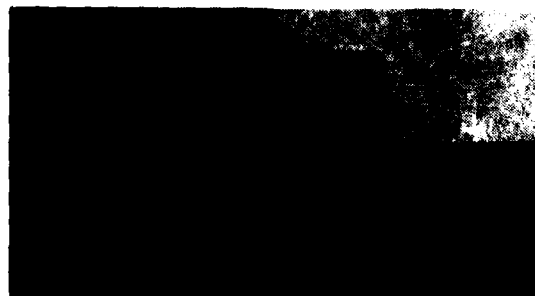


Figure 6. Discoloration stripes.



Figure 7. Bubbles in lining material.

## 4 CONCLUSIONS AND RECOMMENDATIONS

### Specifications

The asbestos-cement pipe met the specifications under which it was procured; however, the epoxy lining failed to meet requirements in some samples.

### Causes of Lining Failure

The epoxy lining in the asbestos-cement pipe failed due to swelling. The swelling apparently occurred as a result of improper proportioning of curing agent in the epoxy mixture at the time of application, or some factor in the curing process, or both.

The variation in the amount of swelling observed in chemical resistance tests indicates that little confidence can be placed in the epoxy lining in any section of pipe with a lining thickness of more than about twice the nominal 12 mil minimum allowed by specifications. Each section of such pipe must be suspect since all samples provided failed two or more chemical resistance weight gain requirements. Only two of the seven linings passed 7-day exposure in distilled water. No sample passed the acidic environment.

Contributing to the failures were several other factors. The great thickness of the lining material and centrifugal casting allowed the development of composition gradients through the film, which resulted in residual stresses at significant levels in the cured material. Swelling of the lining material caused severe strength and hardness losses, which allowed displace-

ment of the material as blisters or section sloughing to relieve the added stress caused by swelling. The diffusion of soil water solutions of sulfates through the pipe material also helped to break down the adhesive bond of the lining to the pipe.

In summary, the failures occurred because of the following factors:

1. Poor proportioning of curing agent to epoxy resin, mixing, and curing, which led to:

- a. Residual stresses
- b. A large amount of swelling
- c. Softening
- d. Strength loss both internally and at the adhesive bond.

2. Environmental effects (high soluble salt content of the soil) which could be tolerated by "good" lining, and possible aggressiveness of the materials carried in the pipe.

### Recommended Solutions

1. If problems occur, remove from service all epoxy-lined asbestos-cement force mains and gravity sewers that have "thick" linings. [In this context, "thick" refers to any lining that is more than double the nominal 12 mil (0.3 mm) thickness.] The removal or replacement process may be done in stages. Screens and filters may have to be cleared often as the pipe lining continues to deteriorate.

2. Replace the pipe with either the "thin" lined epoxy-lined asbestos-cement pipe, or preferably Schedule 40 Polyvinyl Chloride (PVC) pipe of the same nominal diameter. Install the pipe according to the manufacturer's directions. In the diameters involved, PVC and asbestos-cement pipe are about equal in installed cost. PVC is resistant to all of the test reagents in the use temperature range of 40°F (4°C) to 140°F (60°C).

3. Inspect the installation of the pipe more closely. A trip report narrative of an inspection of the failed pipe indicated incomplete finishing of installed pipe at some manholes.

4. In future installations, require closer quality control and inspection of asbestos-cement pipe lining to assure adequacy for the intended service.

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